Technical Report 62

A LOW-TEMPERATURE ADAPTOR FOR THE NORELCO HIGH-ANGLE SPECTROMETER

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O. N. R. Contracts N5ori-07801
N5ori-07858

February, 1953

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Abstract: A simple device for use with the Norelco high-angle spectrometer in maintaining a powder sample at temperatures as low as 78°K is described.

It frequently happens that X-ray powder patterns at low temperatures are needed. The Norelco high-angle spectrometer may be used readily to determine spacings with an accuracy of about 0.0005Å. A simple adaptor has now been devised for using this spectrometer with samples cooled to 78°K. The cryostat is shown schematically in Fig. 1, and consists of a sample holder, an insulated, open chamber for holding liquid nitrogen, and an insulated cover to prevent ice formation near the sample.

The sample holder differs from the usual room-temperature type only in having a ridge on the underside to help form a seal for the liquid nitrogen chamber and a recess on the upper side of the plate. The recess accommodates two strips of mica, which minimize heat transfer from the goniometer to the sample holder. This is accurately machined to bring the upper surface of the sample holder precisely into the same plane as the flat on the goniometer shaft. The liquid-nitrogen chamber was grooved on three of the inside walls, enabling it to be slid onto the sample holder. The insulated cover was constructed with Styrofoam* walls and has two layers of cellophane spaced about 5 mm apart,

*Dow Chemical Company, Midland, Mich.

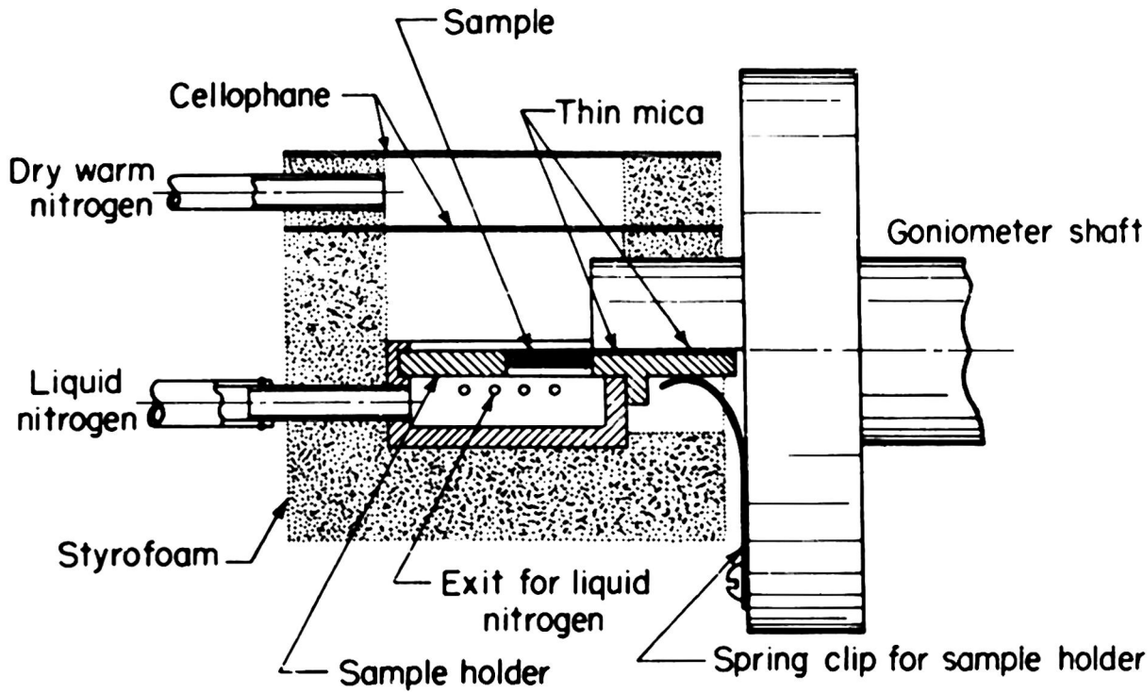

Fig. 1. Low-temperature adaptor for the Norelco high-angle spectrometer.

the lower piece being about 10 mm above the surface of the sample holder.

The sample holder was filled by pressing its polished surface against a polished glass plate and then packing the powder in the resulting cavity from the rear.[1] This forms the surface of the powder into a plane which is level with the surface of the sample holder. The sample was supported by a thin steel plate, and the space between this and the holder sealed with Vinylseal* to avoid liquid nitrogen seeping through to the sample. After the sample holder was mounted on the goniometer, the liquid nitrogen chamber could be slid on, and the joints thoroughly sealed with the Vinylseal. The end of a phosphor bronze spring clip was wedged between the sample holder and the groove in the liquid nitrogen chamber. This was used to press the thermocouple junction tightly against the sample holder about 3 mm from the sample. The insulated cover could then be placed in position to protect the sample and was secured

1) J. Kalnajs, private communication.

* Bakelite Corporation, New York, N.Y.

with friction tape. The temperature of the sample was determined to $\pm$ 2°K.

In order to avoid cooling the large precision gear, a heater of chromel resistance wire was wound around the spring-clip holder of the goniometer, and a thermocouple cemented to the clamp between the heater and the gear. A power input to the heater of about 100 w sufficed to maintain the bulk of the goniometer at room temperature.

Dry nitrogen at room temperature was then passed into the space between the two layers of cellophane, and liquid nitrogen into the lower chamber. The consumption of liquid nitrogen was about 5 ℓ per hour, but this could be reduced by using an insulated connection between a 25-ℓ container and the cryostat. The flow of liquid nitrogen could be adjusted to provide the desired temperature, and about 3 psi of dry nitrogen gas was sufficient to prevent any ice formation under humid conditions.

This device has been successfully used in our investigation of the transition in magnetite,[2] down to 78°K.

The authors wish to thank Professor A. von Hippel for his interest in this work.

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2) S.C. Abrahams and B.A. Calhoun, Technical Report No. 61.